December 31, 1963

Final Summary Report, | February 18, 1962 to December 31, 1963

t: INVESTIGATION OF THE SPACE STORABILITY

OF PRESSURIZING GASES   Final... Report, Feb... 1963

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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Approved:

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PHYSICAL SCIENCES RESEARCH

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ABSTRACT

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Laboratory experiments indicate that a column of lithium borohydride powder will generate hydrogen on demand by reaction with controlled additions of water to an asbestos wick when cobaltous chloride is used as a catalyst and asbestos pulp is interspersed throughout to prevent formation of impermeable, glassy coatings of lithium metaborate.

A comparison is made of computed storage efficiencies for pressurized hydrogen and nitrogen in a system designed to generate hydrogen at about 1.5 atm from lithium borohydride with water added in a controlled fashion by flow through a porous plug. The maximum storage efficiency of such a hydrogen gas generating system, based on its use in an attitude-control system, approaches 27.7 lb-sec of impulse per pound of system weight for a mission requiring a total impulse of 10,000 lb-sec; hydrogen gas and nitrogen gas stored at 1500 psia have values of 16.6 and 33.9 lb-sec per lb, respectively. Thermal decomposition of lithium borohydride or other materials presents no advantage.

A UTHOR

-R. F. Muraca

FINAL SUMMARY REPORT

SRI Project PSU-4000

The Interim Report entitled "Investigation of the Space Storability of Pressurizing Gases," NASA Contract NAS7-105, dated August 1963 describes the major portion of work performed on this contract over the period February 19, 1962 to February 18, 1963.

As a part of this contract, a handbook was to be issued which would include information valuable to engineers designing pressurized-gas systems. A rough draft of the handbook was approved for publication by the program monitors (Messrs. Compitello and Porter). The published volume is entitled "Design Data for Pressurized Gas Systems," and dated November 1963; it is a loose-leaf binder containing 15 sections of data on various topics, including extensive tabulations and graphs of the physical and thermodynamic properties of pressurizing fluids.

The work performed during the period February 19, 1963 to December 31, 1963 consisted largely of the preparation of material for the design data book, and needs no formal reporting here. The succeeding pages of this report summarize work performed on the evaluation of a candidate gas generating system suitable for attitude-control, pressurized-gas supply systems.

STORAGE EFFICIENCY OF LITHIUM BOROHYDRIDE SYSTEMS

Some alternatives to simple pressurization for storage of gases to be used as working fluids for attitude control systems were given in Section VII of the Interim Report on this project (August 1963). Preliminary calculations indicated that if pressurized nitrogen is used for comparison there should be a weight advantage in the use of lithium borohydride as a source of gas for attitude control systems.

INTRODUCTION

Lithium borohydride, $LiBH_4$, is available from commercial sources as a white to grayish microcrystalline powder or lumps. It is stable at room temperature, but the powder must be handled in air with great caution. Generally, its transfer is effected in atmospheres of dry nitrogen or argon (dry box). Water vapor in the atmosphere readily reacts with lithium borohydride:

$$LiBH_4 + 2H_2O = LiBO_2 + 4H_2 \qquad \Delta H = -15.23 \ Kcal \qquad .$$
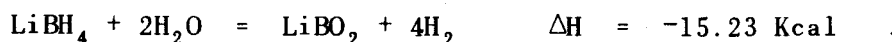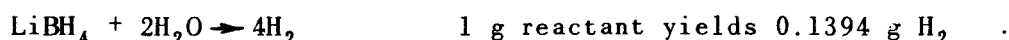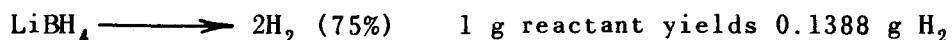
The heat of reaction may cause thermal decomposition and the material may deflagrate in air (especially the powder). Traces of water on combustible material like cellulose generally will react violently with $LiBH_4$; as a result when $LiBH_4$ powder comes into contact with materials like paper or cloth, spontaneous combustion is likely to occur. The properties of lithium borohydride are summarized in Table I.

Table I

PROPERTIES OF LITHIUM BOROHYDRIDE

Formula:	$LiBH_4$
Formula Weight:	21.79
Structure:	ionic, $Li^+ \ BH_4^-$
Crystal Parameters:	orthorhombic, 6.81; 4.43; 7.17
Melting Point:	284°C (decomp.)
Heat of Formation:	44.1 Kcal/mole (Exo.)
Specific Heat:	0.84 cal/g/°C
Solubility:	soluble (with decomposition) in water and alcohols; 4 g/100 g ethyl ether; 21 g/100 g tetrahydrofuran

A cursory survey of the literature indicated that the mode of thermal decomposition is little understood and that only 75% of the hydrogen content of lithium borohydride is readily released by heat. The thermal decomposition is, of course, endothermic; thus the reaction could be readily used to supply hydrogen on command by application of heat. With only 75% recovery of hydrogen from the thermal decomposition of $LiBH_4$, the evolution of hydrogen by reaction with water is competitive:

$$LiBH_4 \longrightarrow 2H_2 \ (75\%) \quad \text{1 g reactant yields 0.1388 g } H_2$$

$$LiBH_4 + 2H_2O \rightarrow 4H_2 \quad \text{1 g reactant yields 0.1394 g } H_2 \quad .$$

Further, it was believed simpler to be able to control the generation of hydrogen by metered additions of water than to control heat input to a relatively nonconductive mass of reactants. Thus, the investigation of the parameters governing the release of hydrogen from $LiBH_4$ was begun.

REACTION OF WATER WITH $LiBH_4$

Available data on the reaction of water with lithium borohydride simply indicated that reaction takes place readily, but no information was available on the smoothness or completeness of reaction. On the other hand, the reaction of sodium borohydride with water has been studied in some detail by Schlesinger, et al.*; the salient point to be made is that the hydrolytic reaction of sodium borohydride is slow, but that it can be accelerated by various substances and notably cobaltous chloride. The effect of the cobalt salt appears to be through route of formation of a black material of empirical composition Co_2B which is formed in the initial stages of the reaction of water with sodium borohydride, and this material serves to catalyze the subsequent reaction.

As a result of the studies, pellets containing 92.5% $NaBH_4$ and 7.5% $CoCl_2$ are offered on the market as "Hydropills" by Metal Hydrides, Inc.; the pills produce hydrogen gas readily upon contact with water. The formation of the catalyst appears to involve the reaction:

$$4CoCl_2 + 8NaBH_4 + 18H_2O = 2Co_2B + 8NaCl + 6H_3BO_3 + 25H_2 \ .$$
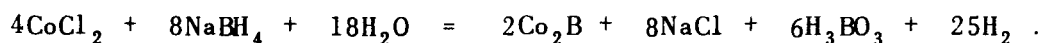
The pills may be used to replace conventional laboratory hydrogen generators.

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* Schlesinger, H. I., Brown, H. C., Finholt, A. S., Gilbreath, J. R., Ho Hoekstra, H. R., and Hyde, E. K., J. Am. Chem. Soc., 75, 215 (1953).

Lithium borohydride, as expected, also is hydrolyzed slowly by water; the hydrolysis is accelerated by cobalt salts analogously to the sodium derivative, and is of interest for possible use as a hydrogen generant in space applications since it offers a reduction in weight of reactants by about 22% over the sodium salt. The weight-volume relationships for the reaction of lithium borohydride with water are summarized in Table II.

Table II

WEIGHT-VOLUME RELATIONSHIPS FOR THE REACTION:
$$LiBH_4 + 2H_2O = LiBO_2 + 4H_2$$

WEIGHT			VOLUME OF H_2 (STP)
$LiBH_4$	H_2O	H_2	
1.000 g	1.654 g	0.3699 g	4.115 liters
0.6048 g	1.000 g	0.2237 g	2.488 liters
2.703 g	4.470 g	1.000 g	11.12 liters
0.2430 g	0.4019 g	0.0899 g	1.000 liters
1.000 lb	1.654 lb	0.3699 lb	65.91 ft^3
0.6048 lb	1.000 lb	0.2237 lb	39.86 ft^3
2.703 lb	4.470 lb	1.000 lb	178.18 ft^3
0.01517 lb	0.02697 lb	0.005612 lb	1.000 ft^3

LABORATORY EXPERIMENTS — WATER

Pellets were prepared from a mixture of 5% anhydrous cobaltous chloride and 95% $LiBH_4$ (Metal Hydrides, Inc.) in an inert atmosphere. It was found that only when excess water was added was 95-98% of the theoretical volume of hydrogen liberated from the pellets. Since the pellets were not prepared from material of the theoretical purity and under conditions in which moisture was strictly excluded, it was reasonable to assume that the yield of hydrogen was essentially quantitative. However, if only a small amount of water was added and the reaction allowed to proceed until hydrogen evolution ceased, further addition of water led to no, or at least a very slow, reaction; the sharply limited or inhibited rate of reaction was attributed to the presence of a tight "cake" of metaborate surrounding each pellet. It appears that the best method for obtaining quantitative generation of hydrogen from such pellets involves treating each pellet with sufficient water to dissolve the $LiBO_2$ which is formed, or to add water to each pellet at such a rate that the concentrated solution of $LiBO_2$ which initially forms is not dehydrated (by unreacted $LiBH_4$) to a slowly-soluble glass. In any instance, however, it appears that the generation of hydrogen by reaction of water with pellets can not be controlled simply and, indeed may be eruptive. A reaction of this type obviously can not be reliably controlled in spacecrafts.
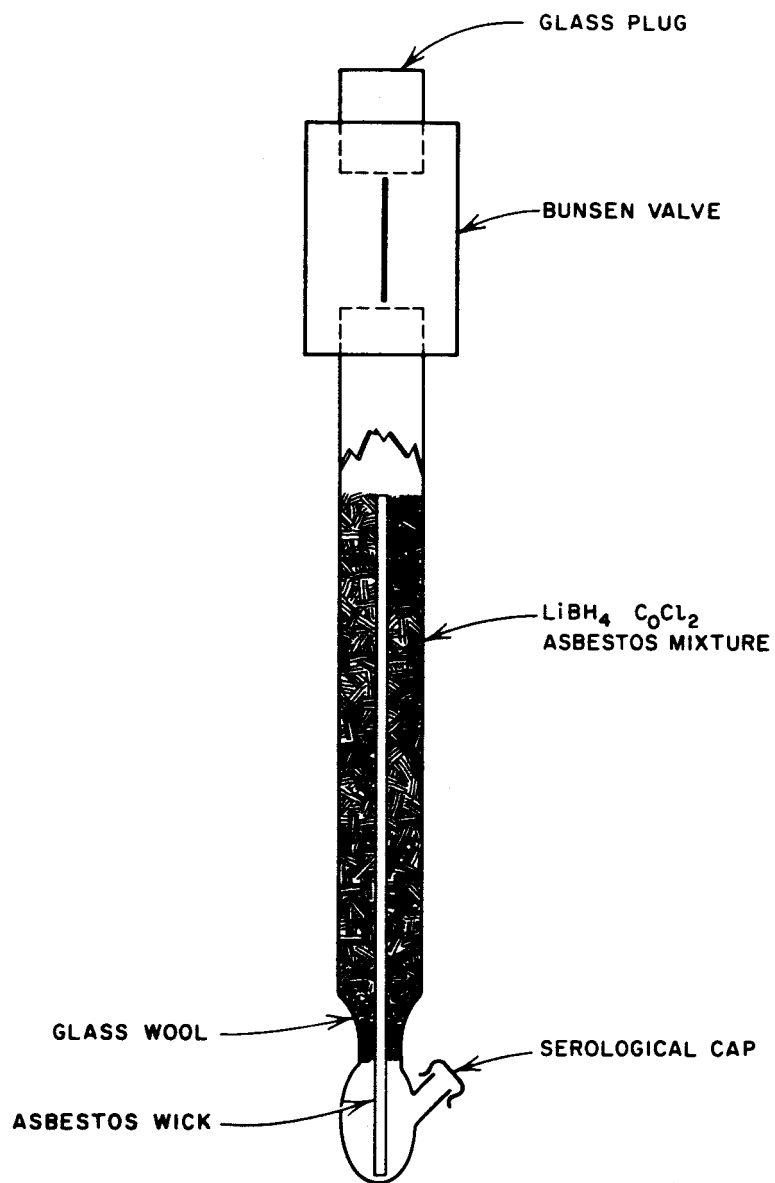
LABORATORY EXPERIMENTS — WATER VAPOR

The reaction of water vapor with a powdered mixture of 95% $LiBH_4$ and 5% $CoCl_2$ was then investigated as a possible alternative route to the controlled formation of hydrogen from $LiBH_4$. The concept guiding the investigation was that water vapor can be generated easily in zero-g and its amount controlled by regulating the volume of water introduced into a system.

Preliminary experiments in which a $LiBH_4$-$CoCl_2$ powder mixture was allowed to have continuous access to water vapor revealed that an essentially quantitative yield of hydrogen could be obtained. In order to investigate the caking problem in intermittent operation, the simple apparatus shown in Figure 1 was constructed. (The filling configuration shown in this figure is the one finally adopted.) As indicated in the figure, the apparatus consists essentially of a reservoir with a side arm closed off by a serological cap, permitting injection of water *ad libitum* by an hypodermic syringe, a column in which powder can be placed, and a Bunsen valve to allow the escape of hydrogen and to inhibit entrance of the ambient atmosphere.

The particular arrangement shown in Figure 1 does not provide means for measurement of the evolved hydrogen; however, replacement of the Bunsen valve with rubber tubing leading to a measuring device made it possible to obtain quantitative measurements of the hydrogen released. The general course of the reaction could easily be followed in instances where the tube was packed with a mixture of $CoCl_2$ and $LiBH_4$ by noting the location of dark zones (black Co_2B). Noting whether the dark material reacted with water provided a convenient check on the degree of decomposition.

In the first group of a series of experiments, water was introduced into the reservoir; the resulting water vapor slowly diffused into the powder column. It was found, as had been found before in experiments with water, that if a continuous supply of water vapor was available and the evolution of hydrogen was continuous, the generation of hydrogen proceeded smoothly and quantitatively. If, however, intermittent operation was attempted by adding only small increments of water to the reservoir, no further reaction could be obtained with subsequent additions of water, especially if considerable time elapsed between water additions.

GLASS PLUG

BUNSEN VALVE

$LiBH_4$ C_oCl_2
ASBESTOS MIXTURE

GLASS WOOL

SEROLOGICAL CAP

ASBESTOS WICK

RB-4000-28

FIG. 1   LABORATORY APPARATUS FOR STUDY OF REACTION
OF WATER ON LITHIUM BOROHYDRIDE

In every case, where hydrogen evolution was inhibited, a well-defined, glassy cake was evident at the bottom of the column of powder. An experiment to avoid formation of the cake was then performed; a central wick of asbestos was used to transport water from the bottom reservoir into the powder mass. As might be expected, intermittent operation, though somewhat possible, was inhibited by formation of a cake around the wick.

The limited success with the use of an asbestos wick prompted a series of experiments in which the 95% $LiBH_4$-5% $CoCl_2$ mixture was mixed with about 5% asbestos pulp and then tamped in the column around an asbestos wick; this is the assembly actually depicted in Figure 1. It was found that intermittent and essentially quantitative generation of hydrogen could be accomplished with this arrangement.

LABORATORY EXPERIMENTS — CONCLUSIONS

A mixture of 95% $LiBH_4$-5% $CoCl_2$ will react smoothly and completely with liquid water or water vapor as long as the generation of hydrogen is continuous. However, special arrangements must be employed when intermittent generation of hydrogen is required, such as for application in space vehicles where the gas pressure must be held between specified limits.

The brief experimental feasibility study reported here was not designed to answer many obviously more detailed questions concerning hydrogen liberation. The desired intermittent operation was achieved by trying only one catalyst at one concentration (although the choice was reasonable from $NaBH_4$ information), using a particular arrangement and relative weight of a specific fiber, and operating only at ambient temperature. Obviously, the interrelation among these variables and the rate of production and yield of hydrogen must be much better delineated before one can be assured with any accuracy that lithium borohydride can be part of a gas storage system for attitude control; but the information gained from the experiments permits design of a gas-generating system which has great possibilities for working successfully.

SYSTEM EVALUATION

The relative merits of gases such as nitrogen, hydrogen, propane, ammonia, etc., in pressurized systems used for attitude control are

generally assessed on the basis of the impulse obtainable per pound of
system weight; when comparing liquefied gases to "permanent" gases, there
are, for example, gains from reduced container weights because of lower
storage pressures, and losses from the lower performance of the liquefied
gases. Because the lithium borohydride-water combination as a source of
pressurized hydrogen for attitude-control systems is completely different
from the ordinary pressurized-gas system, its merits can only be discerned
by a relatively detailed comparison of the anticipated over-all system
weight with those of ordinary systems.

The configuration of what is considered to be a workable $LiBH_4$-H_2O
hydrogen-generating system is indicated schematically in Figure 2. Refer-
ring to this figure, water is contained in an elastomeric bag wholly
within a metal sphere; a pressurizing gas (nitrogen) occupies the ullage.
The opening of the elastomer bag is connected to the inlet of a
magnetically-actuated, spring-loaded valve; when actuated, the valve per-
mits water to pass into a very narrow passageway which terminates at the
face of a dense, porous plug. The water, under pressure, slowly permeates
through the plug and is conducted by capillarity through asbestos fibers
to the reaction zone in which is an intimate mixture of 4.5% $CoCl_2$, 90.5%
$LiBH_4$, and 5.0% asbestos pulp contained in the tubular appurtenance indi-
cated in Figure 2. Hydrogen evolved by the reaction escapes into the
confining cylinder where it is stored under a pressure of 1-2 atmospheres;
when the pressure in the cylinder rises over about 1.5 atmospheres, the
pressure switch releases the magnetically-actuated valve; the water in
the valve duct, porous plug, and asbestos wicks continues to react, but
the pressure in the confining cylinder will not rise much over 2 atmospheres.

The use of a porous plug appears to offer a simple reliable method
for controlling the flow of water into the reaction zone; since the flow
of water through the plug is proportional to pressure and the pressure
gradually diminishes as water is consumed, the rate of flow of water into
the reaction zone is in a measure proportional to the requirements for
smooth generation of hydrogen. This self-regulating feature is one of
the important advantages of the system shown in Figure 2, and it can be
brought to a high degree of development by a careful matching of the
denseness and dimensions of the porous plug with the rate of pressure
drop in the pressurizing sphere and the necessary rate of water flow
for a given configuration of reaction mixture and asbestos wicking.
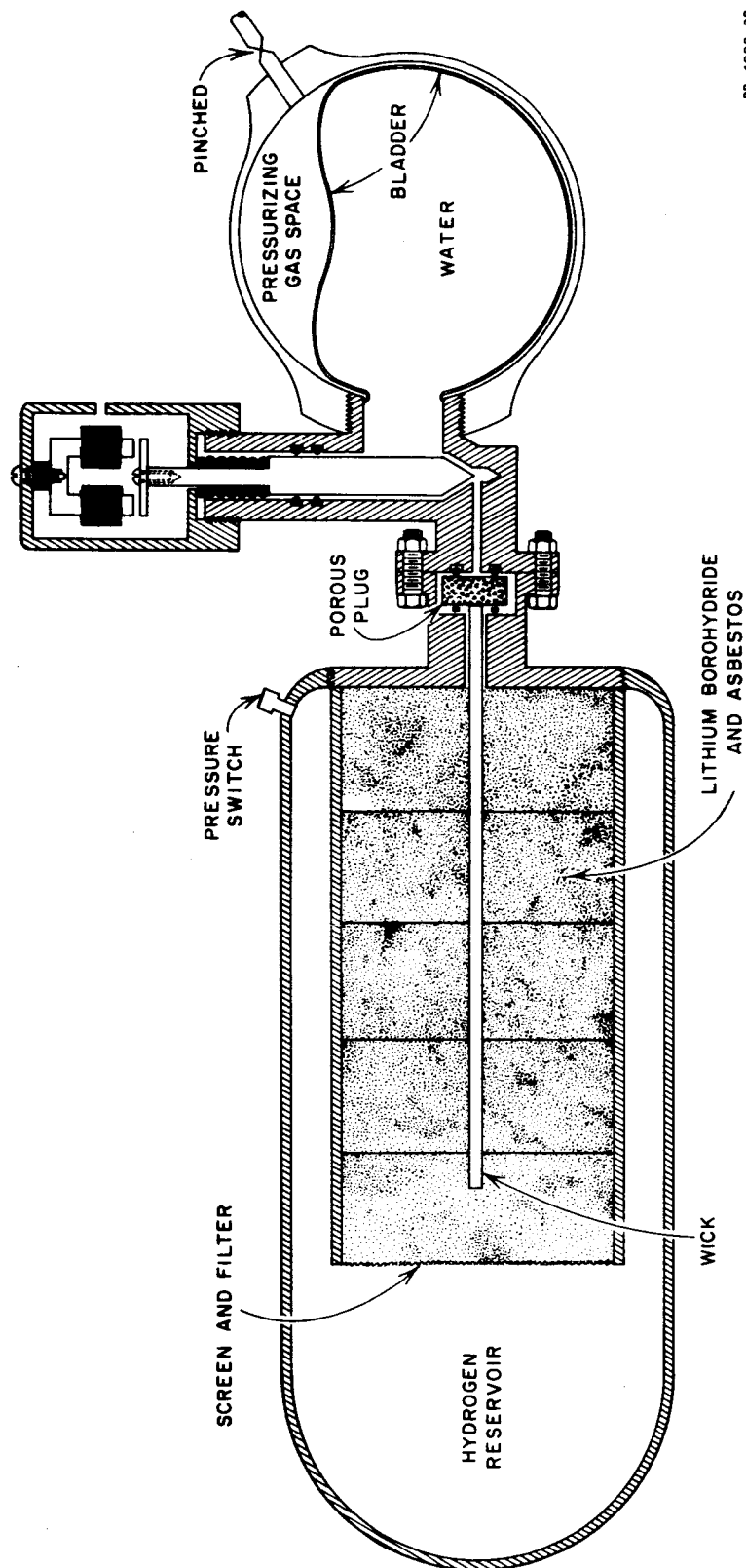
FIG. 2   DIAGRAM OF LiBH₄-H₂O HYDROGEN-GENERATING SYSTEM

It is evident from the above description that the configuration de-
picted in Figure 2 is very light in weight for it is essentially a low-
pressure system; the highest pressure, less than 25 atmospheres, occurs
in the sphere for pressurizing the water required for gas generation.
Thus, the materials of construction can be plastics (as epoxy-glass) or
light metals of extremely thin gage; the thinness of the metals will be
governed largely by the techniques available for fabrication and, of
course, by the need to withstand accelerative forces in the launch phase.

The comparison of the estimated weights for a 1500-psi hydrogen
system, and the $LiBH_4$-H_2O system of Figure 2 are shown graphically in
Figure 3; the data and details for the various systems are recorded in
Tables III, IV, and V. For simplicity, the systems were computed for
missions requiring total impulses of 100, 200, 500, 1,000, 5,000, and
10,000 lb-seconds and with 1,500 psia as the maximum storage pressure
for the permanent gases. Titanium metal 6Al-4V (120,000 psi) was con-
sidered as most suitable for construction of all systems, although it is
conceded that some advantages may be gained by using glass-epoxy storage
vessels; however, the advantage was considered insufficient for the
purposes of these preliminary comparisons.

The theoretical thicknesses of the spherical pressure vessels were
computed by the well-known equation:

$$t = \frac{Pd}{4\sigma - P}$$

where P = pressure, d = inside diameter, and σ = the allowable tensile
stress (120,000 psi for titanium).

No attempt was made to optimize the configuration of the gas storage
container shown in Figure 2; for uniformity of computation, the following
relationships were adopted:

$$L_1 = L_2 \qquad \frac{L_1}{D_1} = \frac{6}{1} \qquad \frac{D_2}{D_1} = \frac{1.5}{1} \ .$$

The Volume, V, of the inner cylinder containing the $LiBH_4$-$CoCl_2$-asbestos
mixture is 10% greater than the volume of the weight needed to provide
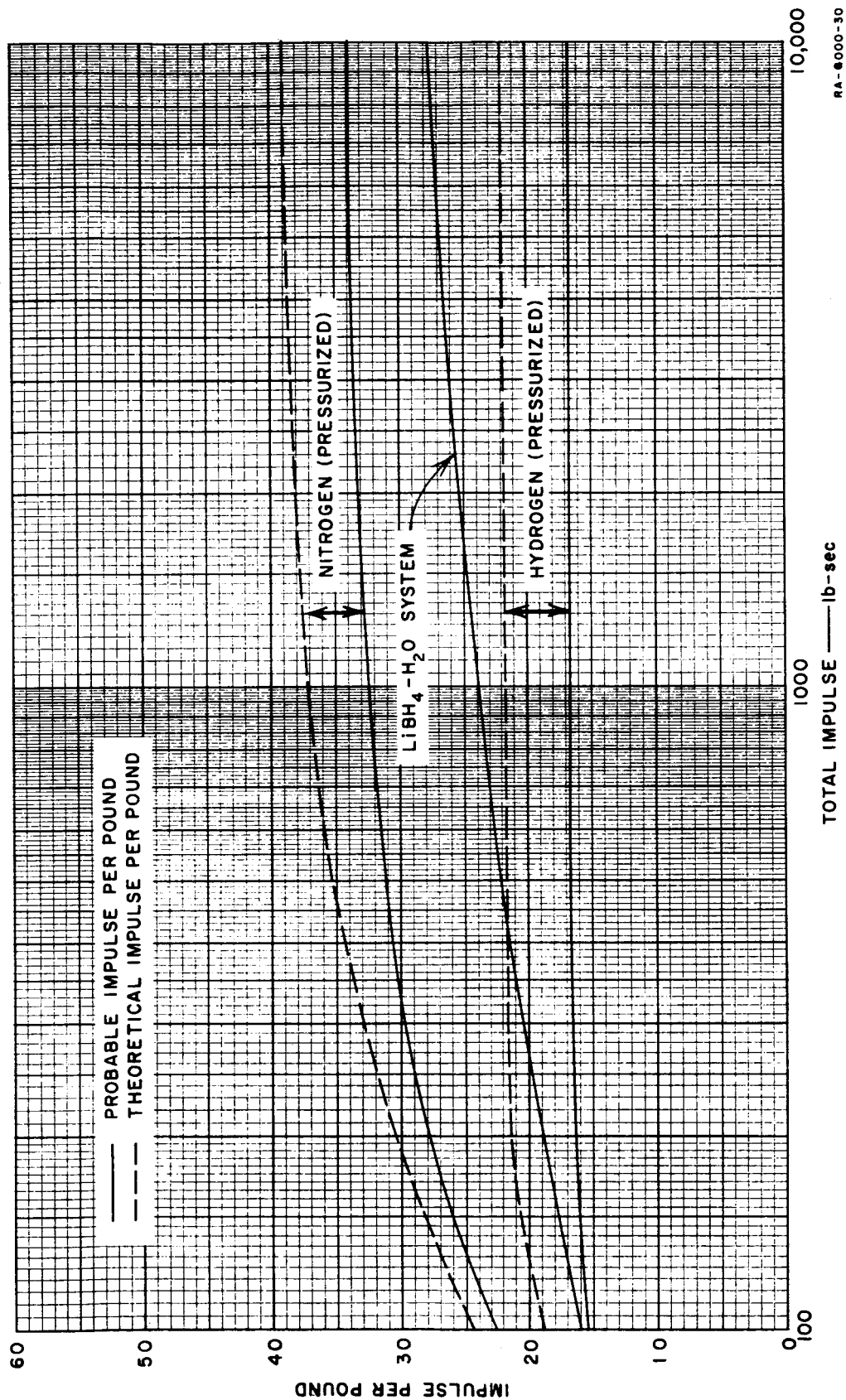
FIG. 3   EFFICIENCIES OF PRESSURIZED GAS STORAGE COMPARED WITH LiBH$_4$-H$_2$O GAS-GENERATING SYSTEM

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Table III

EFFICIENCY OF PRESSURIZED NITROGEN GAS STORAGE

IMPULSE* (lb-sec)	GAS WT (lb)	TANK DIAM† (ft)	THEO. TANK WT§ (lb)	PROBABLE TANK WT§ (lb)	THEO. IMPULSE (per lb)	PROBABLE IMPULSE (per lb)
100	1.45	0.730	2.60	2.97	24.7	22.6
200	2.91	0.922	3.69	4.44	30.3	27.2
500	7.27	1.26	6.91	8.79	35.3	31.1
1,000	14.5	1.58	12.4	16.1	37.2	32.6
5,000	72.7	2.93	58.6	94.0	38.0	33.0
10,000	145.	3.42	110.	147.	39.0	33.9

* Based on nitrogen delivered at 1.5 atm and 538°R to a nozzle with an expansion ratio of 100; sp. ht. ratio = 1.40; calculated I_{sp} = 68.8 sec.

† Based on nitrogen storage at 1,500 psia at 538°R; density = 7.07 lb/ft^3.

§ With added weights: reducing valve = 0.5 lb- control valve = 0.75 lb; fill port = 0.30 lb.

Table IV

EFFICIENCY OF PRESSURIZED HYDROGEN GAS STORAGE

IMPULSE* (lb-sec)	GAS WT (lb)	TANK DIAM† (ft)	THEO. TANK WT (lb)	PROBABLE TANK WT (lb)	THEO. IMPULSE (per lb)	PROBABLE IMPULSE (per lb)
100	0.389	1.18	4.50	6.08	18.8	15.5
200	0.778	1.46	8.50	11.5	21.5	16.3
500	1.946	1.98	21.1	28.5	21.6	16.4
1,000	3.891	2.50	42.5	57.4	21.7	16.5
5,000	19.46	4.25	209.	282.	21.8	16.6
10,000	38.91	5.36	419.	566.	21.8	16.6

* Based on hydrogen delivered at 1.5 atm and 538°R to a nozzle with an expansion ratio of 100; sp. ht. ratio = 1.40; calculated I_{sp} = 257 sec.

† Based on hydrogen storage at 1500 psia at 538°R; density = 0.482 lb/ft^3.

Table V

EFFICIENCY OF LITHIUM BOROHYDRIDE-WATER GENERATING SYSTEM

IMPULSE* (lb-sec)	GAS WT (lb)	WATER WT (lb)	POWDER WT (lb)	POWDER VOL (ft³)	WATER VOL (ft³)	SPHERE DIAM (ft)	SPHERE WT† (lb)	BLADDER WT (lb)	N₂ GAS WT$ (lb)	TOTAL SPHERE WT△ (lb)	D₁ (ft)	L₁ = L₂ (ft)	D₂ (ft)	TOTAL CYL. WT△ (lb)	TOTAL SYSTEM WT# (lb)	THEO. IMPULSE (per lb)
100	0.389	1.739	1.162	0.067	0.028	0.415	0.249	0.025	0.007	2.30	0.250	1.50	0.375	1.64	6.30	15.9
200	0.778	3.479	2.324	0.133	0.056	0.530	0.407	0.039	0.013	4.50	0.314	1.88	0.471	2.58	10.6	18.9
500	1.946	8.696	5.811	0.333	0.139	0.705	0.719	0.072	0.033	10.9	0.427	2.56	0.641	4.78	22.7	22.0
1,000	3.891	17.39	11.62	0.665	0.279	0.890	1.15	0.116	0.066	21.5	0.537	3.22	0.806	7.56	41.9	23.9
5,000	19.46	86.96	58.11	3.33	1.39	1.53	3.39	0.335	0.372	105.	0.919	5.51	1.38	22.2	186.	26.9
10,000	38.91	173.9	116.2	6.65	2.79	1.92	6.40	0.530	0.744	209.	1.158	6.95	1.74	35.2	361.	27.7

* Based on hydrogen delivered at 1.5 atm and 538°R to a nozzle with an expansion ratio of 100; sp. ht. ratio = 1.40; calculated I_{sp} = 257 sec.

† Minimum wall thickness assumed = 0.02 inch.

$ Density = 1.78 lb/ft³ at 25 atm.

△ Plus 5% for miscellaneous additions.

Plus magnetic valve = 0.75 lb; fill port = 0.15 lb; switch = 0.3 lb.

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the required hydrogen; the bulk volume of the mixture was assumed to be 17.5 lb/ft^3; thus:

$$1.10 \text{ (Vol of mix)} = \text{Vol inner cylinder} = \frac{\pi D_1^2}{4} L_1 = V$$

$$\frac{6}{4} \pi D_1^3 = V = 4.7124 \, D_1^3 \quad .$$

The configuration of the gas storage cylinder was assumed to be a cylindrical shell of length L_2 and diameter D_2 with hemispherical ends of radius $D_2/2$.

The bladder confining the water required for the mission (10% in excess) is a 10-mil elastomeric diaphragm of specific gravity 1.00 (wt = 0.052 lb/ft^2). The full volume of water in the pressurizing sphere occupies 75% of the space in the sphere.

The pressurizing gas in the LiBH$_4$-H$_2$O system is nitrogen at 25 atmospheres and the volume is adjusted to give a final pressure of 3 atmospheres.

The pressure of the hydrogen gas collected in the storage reservoir fluctuates between 1 and 2 atmospheres; the powder mixture required was computed on the basis that hydrogen would be liberated quantitatively.

Probable titanium tank weights are 30-40% heavier than calculated from the formula given above; an average of 35% was used.

CONCLUSIONS

As can be seen in Figure 3, the lithium borohydride-water system offers some improvement over pressurized hydrogen gas storage systems for attitude-control systems. A gain of about 50% is realizable over the probable efficiency of pressurized hydrogen, but it is about 18% less than anticipated from pressurized nitrogen gas systems.

No significant gain in storage efficiency can be realized from thermal decomposition of LiBH$_4$ since, as was shown above, the same weight of materials would have to be borne aloft, and the analysis summarized in Table V clearly indicates that the hardware weight is not the dominant factor determining storage efficiency. In fact, if no hardware were involved, the "bare storage efficiency" of LiBH$_4$ itself

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would be 34.4 compared to 33.9 achievable in practice with nitrogen. Table VI summarizes "bare storage efficiencies" for various materials; these values clearly show the superiority of pressurized gases over hydrides. It is interesting to note that water, electrolyzed to "knallgas" has one of the highest "bare storage efficiencies;" however, water vapor itself has an I_{sp} of about 95 (see "Design Data for Pressurized Gas Systems," November 1963).

Table VI

BARE STORAGE EFFICIENCIES* OF VARIOUS MATERIALS
FOR MISSIONS OF 10,000 lb-sec TOTAL IMPULSE

MATERIAL	"BARE STORAGE EFFICIENCY"	REMARKS
$LiBH_4$	34.4	Thermal decomposition to hydrogen
LiH	32.6	Thermal decomposition to hydrogen
BeH_2	46.9	Thermal decomposition to hydrogen; existence of compound is questionable
B_2H_6	56.1	Gaseous decomposition at $T > 300°C$ to $3H_2$
H_2O	85.9	Electrolysis to O_2 and $2H_2$
NH_3	88.3	Decomposition to N_2 and $3H_2$

* "Bare storage efficiency" means the impulse per pound of material obtainable when gas liberated from it by an appropriate method is expanded through a nozzle of 100:1 expansion ratio at an input pressure of 1.5 atm and a temperature of 540°R.

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